



PATENT ABSTRACTS OF JAPAN

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(71)Applicant:

MITSUBISHI CHEM IND LTD

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(72)Inventor:

TAKEDA YOSHIYUKI

YAMADA KENICHI KAWASHIMA OSAMU TERADA YASUSHI

(54) PREPARATION OF TETRAMETHYLUREA

(57) Abstract:

PURPOSE: To prepare the titled compound useful as an intermediate of pharmaceuticals, etc., efficiently, with easy extraction procedure, by keeping a reaction mixture of dimethylamine with phosgene at specific temperature and condition in the presence of a water-insoluble organic solvent, and then separating the mixture into an oil phase and an aqueous phase. CONSTITUTION: Dimethylamine is made to react with phosgene in an aqueous medium in the presence of an alkali (e.g. NaOH, KOH, etc.) to obtain tetramethylurea. In the above process, the reaction mixture is kept at ≥40°C (preferably under atmospheric pressure, and in that case, usually at 40W100°C, preferably 50W90°C) under the condition to allow the water in the reaction mixture to present as a liquid phase (e.g. for 3W120min) in the presence of 0.6W 6pts.wt., based on 1pt.wt. of the tetramethylurea in the reaction mixture, of a water-insoluble organic solvent (e.g. n-heptane, n-hexane, chloroform, etc.) and the resultant mixture is separated into the oil phase containing tetramethylurea and the aqueous phase containing an alkali metal chloride. Tetramethylurea is separated from the oil phase.

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101: 90441a Tetramethylurea. Mitsubishi Chemical Industries Co., Ltd. Jpn. Kokai Tokkyo Koho JP 59 31,752 [84 31,752] (Cl. C07C127/15), 20 Feb 1984, Appl. 82/141,970, 16 Aug 1982; 5 pp. Me₂NCONMe₂ (I) was prepd. by treating Me₂NH with COCl₂ in aq. NaOH or KOH and extd. with org. solvents at > 40°. Thus, 124 g 50% aq. Me₂NH soln. was treated with COCl₂ (75 g/h) in the presence of 264 g 25% aq. NaOH at 0-10° for 1 h and the reaction mixt. was stirred with PhCl at 80° 30 min to give I (from org. layer) with 87% recovery rate compared with 54% at 25°.

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MITU 16.08.82 E(10.4138)	₹ <u>-</u> 3 ₉	## ## ## ## ## ## ## ## ## ## ## ## ##	\$ 55g	
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	V reacting dimentylamine with esence of alkali and keeping in	Presence of water-immiscible re (50.50 day C) under conditions to exists in the liquid phase. I and alkull chloring scale.	is pref. 6.4.6 pts. wt. based on ct. Phosgane is used in amt. of mine, shid alkall in amt. of 6.8.6	methylurga is high (e.g. 87%).
	Dy reach presence argonic solve	n the prese Prore (60.9) mixt. exis	or, Fent is pref. Mixt. Phose Widmine, en	letramethyly
	m in presence . Tale argenit solve	pt in the prese Cormore (60.9) tion mixt. exis oil layer and a	iother golvent is pref. tion mixt, Phosp methylamine, an	rof letramethytu
	Mepa by reacti edium in presence mitcible organic solve	S kept in the prese deg Cormore (50.9) eaction mixt. exis	egon other, anto selvent is pref. ceaction mixt, Phose n dimethylamine, an	ency of letramethyin
	ag propa . medlum in presence rimmiscible organic solve	Xt. Is kept in the prese 1430 deg Cormore (30.9 no resction mixt. exis 8-conts. oil layer and a	om each other organic selvent is pref. In reaction mixt, Phose ed on dimethylamine, an	fficiency of tetra
	wag plopn by reacti ag: medlum in presence voler-immiscible organic solve	nd the present of the	in trom each other. Lof organic selvent is pref. Tree in reaction mixt, Phose based on dimethylamine, an	fficiency of tetra
	e in ag. medium in presence l'of water-immiscible organic solve	tildes 2010n mixt, is kept in the prese piventatiodes Cormore (30.9) et. in the reaction mixt, exis	separation each other, and, of organic selvent is pref. hyl-trea in reaction mixt, Phose mol based on dimethylamine, an	fficiency of tetra
	Dane in ag. medium in presence in a committele organic solve	reaction mixt, is kept in the prese nic solvent at 40 deg Cormore (50.9) water, in the reaction mixt, existing the prese	The and of organic solvent is pref. methylurea in resction mixt. Phosp pts. mol based on dimethylumine, sig	fficiency of tetra
	ahosgane in aq. medium in presence oresence of water-immiscible arganic solve	Lead-College 10 to the present of th	The anti of organic solvent is pref. Chametrylures in resolion mixt. Phosp Of pix. mot based on dimetrylamine, an	fficiency of tetra
	used present by 19. medium in pres 18r-immitatible organ	2328	terramethylures in reaction of the prof. 0 4.6 pis. wt. based on 0.5.2 pis. mol based on dimethylamethylures in reaction mixt. Phaseant is used in amt. of equives.	(dep Dwg. No. 0/0)

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